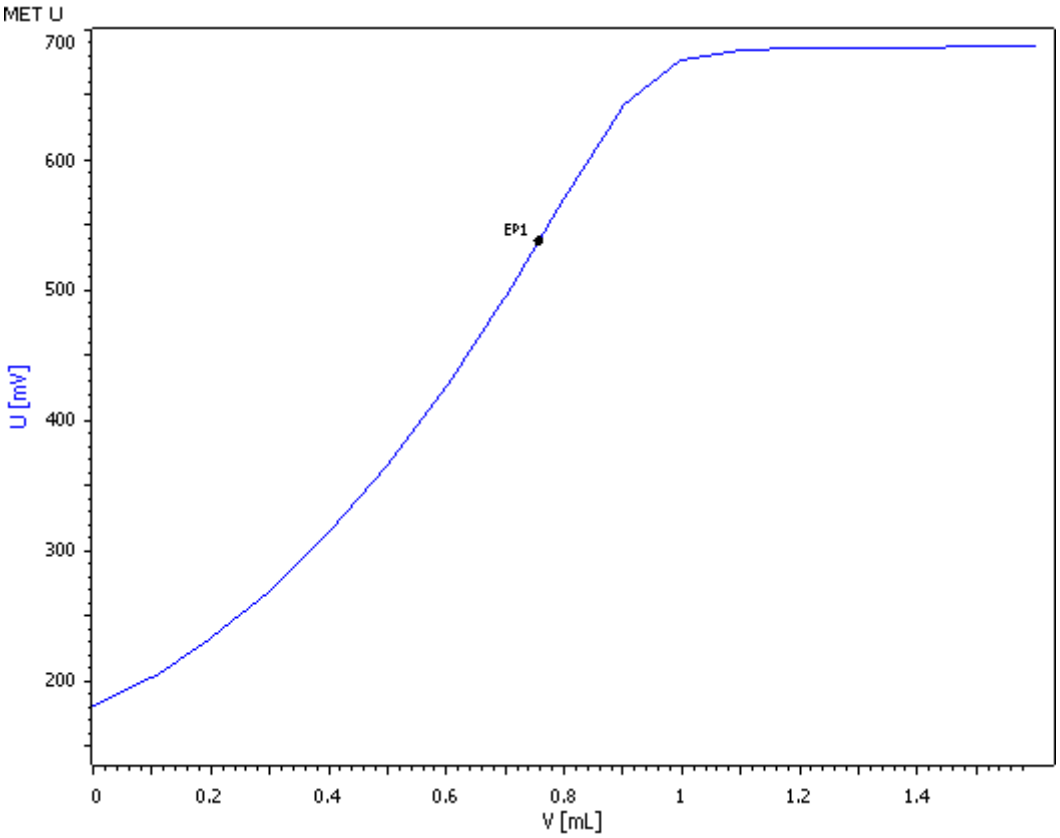
Titration Application Note T–80

Determination of iron in cement by photometric titration



This Application Note describes the digestion of a cement sample and the photometric determination of iron according to DIN EN 196-2 by using the Optrode at 610 nm. Sulfosalicylic acid was used as indicator and EDTA as titrant.

Metrohm

Method description

Sample

Portland-cement

Sample preparation

Approximately 2.5 g cement are mixed with 1 g ammonium chloride and thoroughly brayed with a glass rod. Then, 10 mL conc. HCl and 0.5 mL conc. HNO_3 are added. The mixture is heated in a water bath for 30 minutes. The pasty mixture is diluted with a small amount of dist. H_2O and quantitatively filtered through a coarse-pored filter (595½) into a 500 mL volumetric flask. The filter is well rinsed with hot dist. H_2O. After cooling, the combined filtrate and washings are made up to the mark with dist. H_2O (sample solution).

Configuration

905 Titrando	2.905.0010
800 Dosino	4 x 2.800.0010
Dosing unit 5 mL	2 x 6.3032.150
Dosing unit 10 mL	1 x 6.3032.210
Dosing unit 50 mL	1 x 6.3032.250
802 Rod Stirrer incl. propeller	2.802.0020
814 USB Sample Processor	2.814.0030
Sample rack 12x250 mL	6.2041.310
Sample beaker 250 mL glass	6.1432.320
Optrode (λ = 610 nm)	6.1115.000
Ecotrode plus	6.0262.100
Electrode cable	6.2104.020

Solutions

Titrant	c(Na_2EDTA) = 0.05 mol/L
Ammonium chloride (NH_4Cl)	CAS 12125-02-9
Hydrochloric acid (HCl), conc.	CAS 7647-01-0
Nitric acid (HNO_3),conc.	CAS 7697-37-2
Calconcarboxylic acid indicator solution (stable only one day!)	50 mg carboxylic acid in 100 mL of c(NaOH) = 0.1 mol/L.
w(sulfosalicylic acid) indicator solution	4 g in 100 mL dist. H_2O
Standard solution 6.2301.070	c($CaCl_2$) = 0.1 mol/L Metrohm
c(NaOH) = 2 mol/L	CAS 1310-73-2
c(NaOH) = 1 mol/L	CAS 1310-73-2
c(NaOH) = 0.1 mol/L	CAS 1310-73-2
Ammonium hydroxide	c(NH_4OH) = 25%
Glycine	CAS 56-40-6

Analysis

Titer

2 mL $CaCl_2$ standard are pipetted into a sample beaker, 1.0 mL calconcarboxylic acid indicator, 120 mL dist. H_2O and 4 mL c(NaOH) = 2 mol/L are added. The solution is titrated immediately with c(Na_2EDTA) = 0.05 mol/L.

Sample

25 mL of the sample solution are pipetted into a sample beaker and diluted with dist. H_2O to approx. 120 mL. Then, 0.5 g glycine and 1 mL of sulfosalicylic acid indicator are added. After adjustment to pH 1.5 with 25% NH_4OH, the solution is heated to 50 °C in a water bath to bring glycine into solution. After cooling to room temperature, the mixture is titrated with c(EDTA) = 0.05 mol/L from reddish violet to faintly yellow.

Parameters

	Titer/sample
Titration mode	MET U
Stirring rate	8
Pause (s)	30
Signal drift (mV/min)	50
Min waiting time (s)	0
Max waiting time (s)	26
Volume increment (mL)	0.1
EP criterion (mV)	30
EP recognition	all
Wavelength Optrode (nm)	610

Calculations

$$\% Fe_2O_3 = \frac{V_{EP1} \times c(EDTA) \times t \times 500 \times 159.69 \times 100}{\text{sample size} \times \text{sample vol.} \times 1000 \times 2}$$

V_{EP1}	Consumption of titrant in mL
c(EDTA)	Concentration of titrant in mol/L
t	Titer of the titrant (dimensionless)
500	Dilution factor in mL (here 500 mL)
159.69	Molecular weight of Fe_2O_3 in g/mol
100	Calculation factor for %
Sample size	Weight of sample in g used to prepare the sample solution (here 2.5 g)
Sample vol.	Volume of sample solution used for the determination in mL
1000	Correction factor for L
2	Stoichiometric factor

Results

Fe_2O_3 in %	2.39 ± 0.035 (n = 6)

Metrohm